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Passive Corrosion Behavior of Alloy 22

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Abstract – Alloy 22 (N06022) was designed to stand the most aggressive industrial applications, including both reducing and oxidizing acids. Even in the most aggressive environments, if the temperature is lower than 150°F (66°C) Alloy 22 would remain in the passive state having particularly low corrosion rates. In multi-ionic solutions that may simulate the behavior of concentrated ground water, even at near boiling temperatures, the corrosion rate of Alloy 22 is only a few nano-meters per year because the alloy is in the complete passive state. The corrosion rate of passive Alloy 22 decreases as the time increases. Immersion corrosion testing also show that the newer generation of Ni-Cr-Mo alloys may offer a better corrosion resistance than Alloy 22 only in some highly aggressive conditions such as in hot acids.

I. INTRODUCTION

Alloy 22 (N06022) belongs to the corrosion resistant family of Nickel-Chromium-Molybdenum (Ni-Cr-Mo) alloys. The required composition and mechanical properties of this family are described in ASTM B575 [1]. The grandparent alloy of this family was the cast version of Alloy C first introduced in the market in the 1930s. In the 1960s, the wrought Alloy C-276 (N10276) was developed by basically eliminating the impurities of the cast Alloy C. Alloy C-276 contains approximately 16% Cr, 16% Mo, 4% W and some residual Fe (Table I). Later, Alloys C-4 (N06455) and C-22 (N06022) appeared as modified versions of C-276. In the 1990s, Alloys 59 (N06059), C-2000 (N06200), MAT 21 (N06210) and Inconel 686 (N06686) were designed based on the good performance of Alloy 22, basically by increasing the amount of alloying elements such as Mo and Cr. Alloy 625 (N06625) can also be considered a member of the Ni-Cr-Mo alloy family even though it has less Mo (approx. 9%) (Table I). Many times, Inconel 625 is preferred for high temperature applications rather than for aqueous solutions applications. This series of Ni-Cr-Mo and other commercial alloys provide a range of corrosion resistance materials for designers and engineers to select relevant to the corrosiveness of the service environment. A less corrosion resistant alloy can be selected for a less aggressive range of environments, and this is typically accompanied by lower costs.

Alloy 22 (N06022) was first introduced in the market in the earlier 1980s. It was designed based on the concept of the atomic percent factor (APF), which reflects the opposing role of Cr to that of Mo and W in reducing and oxidizing acidic solutions. A reducing solution has a redox potential with a cathodic current controlled by

hydrogen evolution reaction (HER) and an oxidizing solution has a redox potential controlled by a cathodic reaction with a potential higher than HER (e.g. Fe³⁺ or nitric acid). The APF was described as:

$$APF = \frac{4Cr}{(2Mo + W)}$$

Where the symbols represent weight percentage of the element in the alloy. In oxidizing conditions such as ASTM G 28A, the higher the Cr content (i.e. the higher the APF) the lower the corrosion rate. In reducing conditions such as boiling hydrochloric acid, the higher the Mo and W contents (i.e. the lower the APF) the lower the corrosion rate. The ultimate versatility of an alloy that can be used both in oxidizing and reducing conditions would be a chemical composition that would yield a APF of 2.5 to 3.3. The APF of N06022 is 3.03.

Alloy 22 is also resistant to localized corrosion and stress corrosion cracking in chloride containing environments [2]. As a result, Alloy 22 was selected to build the external shell of the high level nuclear waste containers for the repository in Yucca Mountain [3].

In most applications, the general corrosion rate of Alloy 22 is exceptionally low due to the passive film that forms on its surface when it enters in contact with the various environments. Moreover, when Alloy 22 is immersed in a given electrolyte, the corrosion rate decreases as a function of time due to the development of the passive film, which appears to become more impervious as time increases.

This paper presents a review of the published data and analyzes the general corrosion behavior of Alloy 22 in a variety of environments including simulated concentrated

waters, concentrated brines and acids both as a function of temperature and immersion time.

TABLE I. Approximate Chemical Composition (in wt %) of Ni-Cr-Mo Alloys (listed in alpha-numerical order by UNS number)

Alloy	Ni	Cr	Mo	W	Fe	Other
N06022	57	22	13	3	3	2.5Co*,0.35V*
N06059	60	23	16	-	<1	-
N06200	59	23	16	-	-	1.6Cu
N06210	60	19	19	-	-	1.8Ta
N06455	65	16	16	-	3Fe*	0.7Ti, 2Co*
N06625	62	21	9	-	5*	3.7(Cb+Ta)
N06686	46	21	16	4	5	-
N08825	42	22	3	-	31	2Cu, 1Ti
N10276	57	16	16	4	5	2.5Co*,0.35V*
* Maximum						

II. TESTING METHODS FOR GENERAL OR UNIFORM CORROSION

Uniform corrosion or general corrosion is the “corrosion that proceeds at about the same rate over a metal surface” as defined by the ASTM Standard G 15 [4]. The corrosion rate of metals such as Alloy 22 is influenced by metallurgical and environmental factors. These include surface condition, temperature, pH of the electrolyte in contact with the alloy, exposure time, etc. In most conditions Alloy 22 will remain passive, that is, a protective Cr_2O_3 will form on the surface slowing down the dissolution rate of the underlying metal. As the exposure time to the corroding environment increases the general corrosion rate decreases. This is a well-established fact for passive materials, including Alloy 22.

The corrosion rate is generally calculated in the industry by weight (mass) loss [5,6]. Guidelines for mass loss corrosion rate determination are given in the ASTM Standard G 31 and G 1 [4]. The measurement of corrosion rate by weight loss (WL) is more accurate when the environment is aggressive and the mass losses are easily measured. In the mass-loss procedure, pre-weighed coupons of the alloy to be tested are exposed to the corrosive environment for a given period of time. At the end of the testing time, the coupons are cleaned, dried and weighed. The corrosion rate is calculated dividing the mass loss by the surface area of each coupon (A), the exposure time (t) and the density of the alloy (d).

$$CR \left(\frac{\mu\text{m}}{\text{year}} \right) = \frac{8.76 \cdot 10^7 (W_i - W_f) (g)}{A(\text{cm}^2) \cdot t(h) \cdot d(g \cdot \text{cm}^{-3})}$$

where W_i and W_f are the initial and final mass of the coupon in grams.

The corrosion rate can also be calculated using electrochemical methods such the polarization resistance (PR) method described in ASTM G 59 and G 102 [4-6]. Each of these polarization resistance tests lasts approximately four minutes. An initial potential of 20 mV below the corrosion potential (E_{corr}) is ramped to a final potential of 20 mV above E_{corr} at a rate of 0.167 mV/s. The Polarization Resistance (R_p) is defined as the slope of the potential (E) vs. current density (i) at $i = 0$. The corrosion current density, i_{corr} , is related to the polarization resistance by the Stern–Geary coefficient B

$$i_{\text{corr}} = 10^6 \cdot \frac{B}{R_p}$$

where

$$B = \frac{b_a \cdot b_c}{2.303(b_a + b_c)}$$

where b_a and b_c are the anodic and cathodic Tafel slopes

$$i_{\text{corr}} = \frac{1}{R_p} \times \frac{b_a \cdot b_c}{2.303(b_a + b_c)}$$

The corrosion rate can then be calculated using the Faraday equation

$$CR(\mu\text{m} / \text{yr}) = k \frac{i_{\text{corr}}}{d} EW$$

Where k is a conversion factor ($3.27 \times 10^9 \text{ nm} \cdot \text{g} \cdot \text{A}^{-1} \cdot \text{cm}^{-1} \cdot \text{yr}^{-1}$), i_{corr} is the corrosion current density in A/cm^2 (calculated from the measurements of the resistance to polarization, R_p), EW is the equivalent weight, and d is the density of Alloy 22 ($8.69 \text{ g}/\text{cm}^3$). Assuming an equivalent dissolution of the major alloying elements as Ni^{2+} , Cr^{3+} , Mo^{6+} , Fe^{2+} , and W^{6+} , the EW for Alloy 22 is 23.28 (ASTM G 102) [4].

In the calculations reported here the linear fits were constrained to the potential range of 10 mV below E_{corr} to 10 mV above E_{corr} . During the fitting of the data to calculate the polarization resistance (R_p), the potential or independent variable (E) was plotted in the X-axis. The Tafel constants, b_a and b_c , were assumed to be $\pm 0.12 \text{ V}/\text{decade}$.

III. THE UNIFORM CORROSION PROCESS

The corrosion rate or the thinning of a structure in time is the result of a series of complex reactions on the metal surface. The actual rate at which corrosion proceeds could be activation or diffusion (concentration) controlled. For the long times involved for the Alloy 22 proposed containers, time for diffusion should not be limiting and the corrosion rate can be easily assumed to be activation controlled, that is, the thinning of the container will be controlled by the reactions occurring at the metal/corrosive environment interface and not by the arrival or removal of species to the surface. Nevertheless, since Alloy 22 develops an impervious passive film on the surface, which delays the reaction of the base metal (substrate) with the environment, diffusion processes though this protective film can also play an important role in the overall corrosion process.

IV. THE PASSIVE FILM ON ALLOY 22

Alloy 22, like many other corrosion-resistant alloys, forms a stable chromium (III) oxide (Cr_2O_3) film on the surface in most industrial environments. The presence of this oxide film limits further dissolution of the alloy into the environment [7,8]. Depending on the environment (e.g. solution composition, pH and temperature) the overall oxide scale may have multiple compositions and layers; however, in all of the tested conditions, a protective inner Cr_2O_3 layer was always present (an outer Ni-rich layer is often also present) [7,8]. The Cr(III) oxide layer is thin (<10 nm thick) and may be the key resistant barrier between the alloy and the environment, at least in short-term experiments. The protective oxide film and any distinct layers grow until steady-state thicknesses are achieved, especially for the inner or rate-limiting Cr_2O_3 rich layer. In theory, the thickness of the inner layer would not change further in time. Specimens exposed for a few days to multi-ionic electrolyte solution had similar oxide thickness as specimens exposed for over five years to the same electrolytes [8].

Even though the thickness of the protective oxide film may reach a steady state value in time, the protectiveness of the film may still increase in time due for example to the annihilation of defect density within the film [8,9-11]. The protective oxide film will be always present on Alloy 22, unless this film is removed mechanically or chemically. Mechanical post-closure removal may be a consequence of seismic activity. The oxide film can be removed chemically for example in hot hydrochloric acid solutions or in pickling solutions, e.g. mixtures of nitric acid and hydrofluoric acid.

V. CORROSION BEHAVIOR OF ALLOY 22 IN MINERAL ACIDS

One of the most aggressive reducing acids is hydrochloric acid (HCl). Figure 1 shows the corrosion rate of Alloy 22 (and other newer Ni-Cr-Mo alloys) in 10% HCl as a function of the temperature using the weight loss method. Alloy 22 is highly resistant to corrosion in low temperature HCl because the passive oxide film is stable in these conditions. However, when the temperature is raised above 150°F (66°C) Cr_2O_3 becomes less protective and the corrosion rate of Alloy 22 starts to increase due to the active dissolution of the metal. It is likely that HCl reacts with Cr_2O_3 to form soluble complexes of chromium and chloride (CrCl_3). Basically, for Alloy 22 (and other Ni-Cr-Mo alloys) in hydrochloric acid, the burden of resistance against corrosion is shifted from Cr to Mo when the temperature is raised from cold to hot. When the passivity is broken in hot HCl, the corrosion rate of the newer Ni-Cr-Mo alloys are lower because they contain higher amount of Mo, a beneficial alloying element for HCl service [12-15].

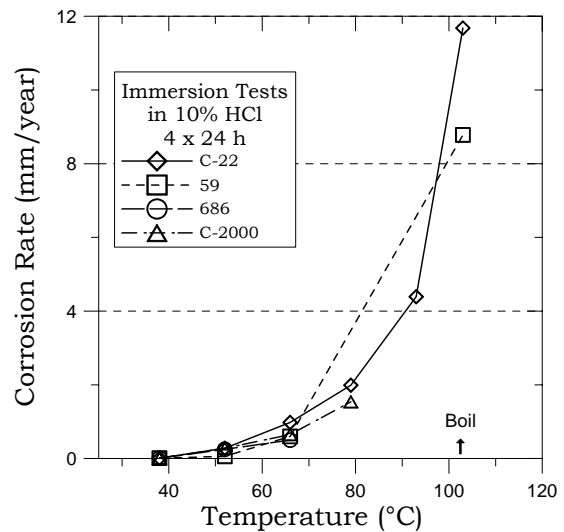


Fig. 1. Corrosion rate of Ni-Cr-Mo Alloys in HCl [16]

Figure 2 shows the corrosion rate of Alloy 22 (and other Ni-Cr-Mo alloys) in 50% H_2SO_4 as a function of the temperature (weight loss method). A similar behavior as in the case of HCl is observed, that is, while the alloy maintains its passivity (i.e. not active corrosion) at temperatures below 80°C, the corrosion rate is low and difficult to detect using short-term immersion tests. Figure 2 also shows that the lowest corrosion rate corresponded to alloy C-2000 because this alloy contains a small amount of copper (Cu) a beneficial alloying element for H_2SO_4 service [13-15]. The comparison of Figs. 1 and 2 show that HCl is much more aggressive than H_2SO_4 towards the studied Ni-Cr-Mo alloys. Data in Figs. 1-2

also show that only under the most aggressive conditions the corrosion rates of the newer Ni-Cr-Mo alloys (N06659, N06200 and N06686) could be lower than the corrosion rate of Alloy 22, and this difference is at the utmost a factor of 2. At temperatures lower than 80°C the corrosion rate of all the Ni-Cr-Mo alloys seem undistinguishable from each other (Figs. 1-2).

Other acids such as hydrobromic, phosphoric, acetic, etc. are much less corrosive towards Ni-Cr-Mo alloys [14] than the acids reported here such as HCl and H₂SO₄.

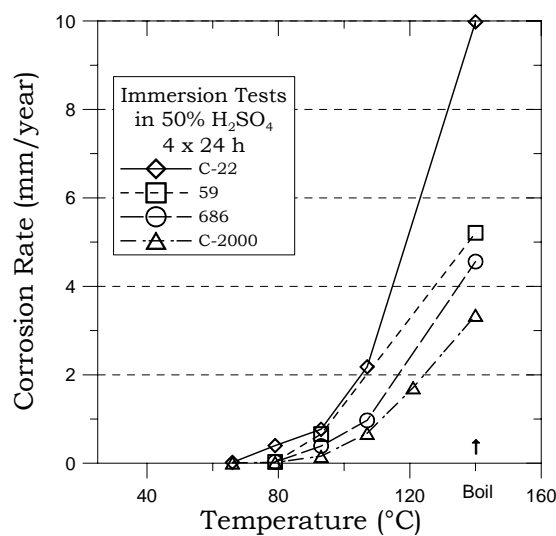


Fig. 2. Corrosion rate of Ni-Cr-Mo Alloys in H₂SO₄ [16]

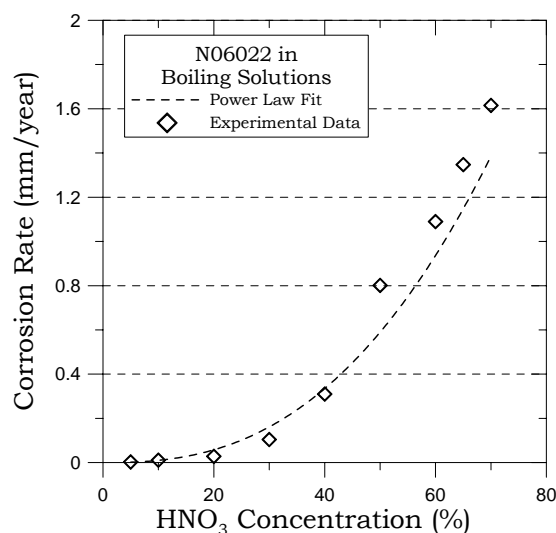


Fig. 3. Corrosion rate of Ni-Cr-Mo Alloys in HNO₃ [16]

Fig. 3 shows the corrosion rate of Alloy 22 in boiling nitric acid as a function of the acid concentration using the weight loss method. The maximum corrosion rate of Alloy 22 for example in 40% acid is less than 0.4 mm/year (40% HNO₃ is 7.9 M or 10.6 m and boils at

approx. 112°C). Figure 3 also shows that, at the same temperature, the corrosion rate of Alloy 22 in HNO₃ is much lower than in HCl (Fig. 1) or H₂SO₄ (Fig. 2) solutions. Fig. 4 shows that HNO₃ even reduces the corrosion rate of Alloy 22 when in the presence of HCl. For example, for a 10% HCl solution, the corrosion rate drops one order of magnitude when nitric acid is added to a ratio HNO₃/HCl of 2 or higher. Fig. 4 also shows that when the ratio of nitric to hydrochloric is lower than 2 the corrosion rate of Alloy 22 is not substantially suppressed. The inhibiting effect of HNO₃ on the dissolution of Alloy 22 in HCl solutions has also been determined using titration experiments [17].

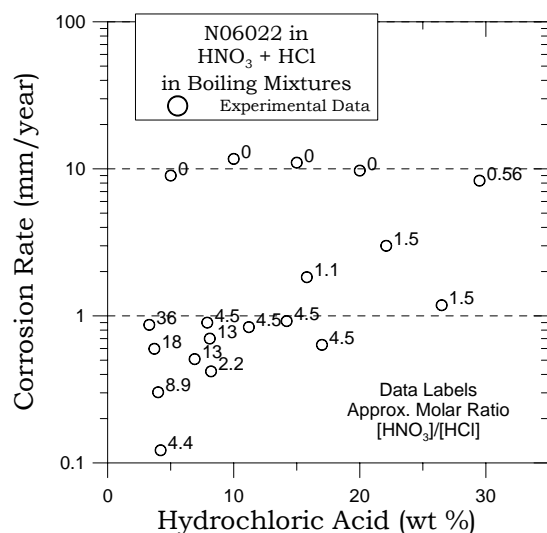


Fig. 4. Corrosion rate of Ni-Cr-Mo Alloys in HCl and HNO₃ Mixtures using the Weight-loss Method [16]

There are two main modes of general corrosion of chromium containing alloys such as Alloy 22. These are the (1) active corrosion and the (2) passive corrosion. Active corrosion generally occurs under reducing conditions in a medium such as hot HCl or hot dilute H₂SO₄ acid. During active corrosion the chromium oxide passive film is not stable and the alloy relies on intrinsic properties of the alloying elements (such as molybdenum) for corrosion resistance. In general, active corrosion does not occur on Alloy 22 at ambient temperatures regardless of the aggressiveness of the electrolyte (acid). Typically when active corrosion occurs, the corrosion rate increases steadily as the temperature increases. For the same environments (e.g. Fig 1), when the temperature is below 66°C the corrosion rate is practically nil, using the same testing method of weight-loss. That is, there is a cut-off in corrosion rate behavior vs. temperature at a certain threshold temperature value. This threshold temperature depends on the testing medium. This cut-off corrosion behavior is due to a change in the corrosion mode from active to passive.

In most applications, Alloy 22 remains in the passive state; that is, the oxide film is stable and provides the barrier of protection between the alloy and the environment. Even under anodic applied potentials in chloride solutions of pH 1 and up to 85°C, Alloy 22 remained passive [18]. The environments that may be related to the proposed repository conditions are mild compared to some industrial applications such as the handling of acids. The repository environments are predicted to be multi-ionic near neutral aerated salt solutions containing mainly chloride, nitrate, sodium and potassium. Alloy 22 has a passive corrosion behavior in such environments. The degree of passivity will largely depend on the immersion time. As the time increases, the passive current (dissolution rate) always decreases.

VI. PASSIVE BEHAVIOR OF ALLOY 22 IN CONCENTRATED SALT SOLUTIONS

In the previous section it has been reported that Alloy 22 is not highly prone to corrosion even when exposed to hot concentrated mineral acids. It would then be expected that the corrosion rate of Alloy 22 in lesser acidic salt solutions would be even lower. Experimental evidence shows that near neutral concentrated multi-ionic solutions the corrosion rate of Alloy 22 is lower than in mineral acids by several orders of magnitude [19]. After 5-year immersion in aqueous electrolyte solutions simulating concentrated ground waters from pH 2.8 to 10 at 60°C and 90°C, the corrosion rate of Alloy 22 was in the order of 10 nm/year and lower [20-21]. For short-term the corrosion rate of Alloy 22 was reported to be in the order of 1 $\mu\text{m}/\text{year}$ and lower [22]. Results were from different sources and for several electrolyte solutions including simulated acidified water and simulated concentrated water [22]. It has also been reported that the corrosion rate of Alloy 22 after an 8-week immersion in basic saturated water pH 12 from 60°C to 105°C was between 76 nm/year and 305 nm/year [22]. Short-term immersion of Alloy 22 in 35% MgCl_2 (~6 *m*) solutions produced a maximum corrosion rate of approximately 10 $\mu\text{m}/\text{year}$ at 120°C using the Electrochemical Impedance Spectroscopy Method [23]. This higher value in the corrosion rate was probably caused by a disruption of passivity in Alloy 22 in presence of the acidic MgCl_2 brine [23]. Figure 5 shows the short-term corrosion rate of Alloy 22 in 10-18 *m* CaCl_2 brines between 110°C and 160°C using the Polarization Resistance Method. The corrosion rate values of Alloy 22 in Fig. 5 are slightly lower but in the same order as the ones reported by Dunn et al. [23]. The environment at the proposed repository site is never expected to evolve to become a single substance solution (such as a CaCl_2 brine).

VII. EFFECT OF TIME ON THE CORROSION RATE OF ALLOY 22

As the immersion time increases the corrosion rate of Alloy 22 tends to decrease due to the formation of a more compact protective passive film [9-11,18,24]. After a certain time, the passive film may stop growing; however, the passive current keeps decreasing in time probably due to the continuous annihilation of defects in the film [9-11]. Fig. 6 shows the effect of the immersion time on the corrosion rate of Alloy 22 specimens immersed for over 8 months in a naturally aerated concentrated brine of 6 *m* NaCl + 0.9 *m* KNO_3 at 100°C. Both for the polished specimen and for the specimen containing a black annealing oxide film on the surface, the corrosion rate was in the order of 20 nm/year for the longest immersion time (Fig. 6).

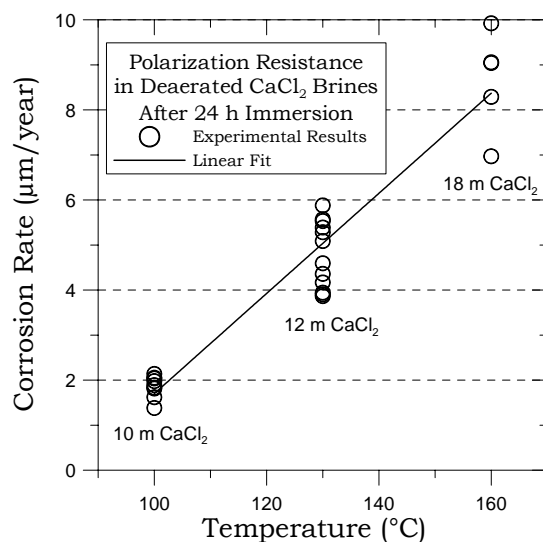


Fig. 5. 24-h Corrosion Rate of Alloy 22 in CaCl_2 Brines [25]

VIII. FINAL REMARKS

The corrosion rate of Alloy 22 is dependent on the temperature especially when corrosion occurs in the active condition. The corrosion rate of Alloy 22 is high only in hot reducing acids. For oxidizing acids (Fig. 3) and highly concentrated brines (Fig. 4) the corrosion rate of Alloy 22 is low. Nevertheless, it is never expected that the environment in Yucca Mountain would evolve to have a pH of less than zero or be constituted by a pure substance (such as HNO_3 or CaCl_2). Moreover, below approximately 60°C Alloy 22 would remain in the passive mode independently of the aggressiveness of the electrolyte solution. The nuclear waste containers proposed for Yucca Mountain are expected to have a temperature below 60°C at times of 8,000 year or longer [26]. That is, for most of their emplacement time, the

temperature will be low enough for the alloy to remain in the permanent passive state.

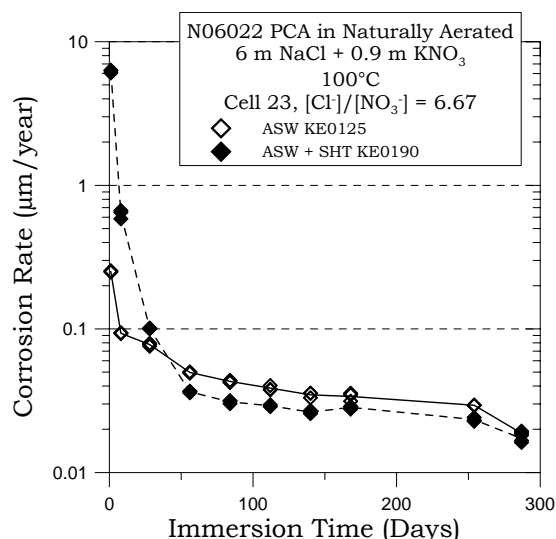


Fig. 6. Corrosion Rate vs. Immersion Time

IX. SUMMARY AND CONCLUSIONS

Alloy 22 has been designed to withstand the most aggressive industrial applications.

Since the commercial introduction of Alloy 22 in 1983, a newer generation of Ni-Cr-Mo alloys has appeared.

The newer alloys were mostly designed to be an improvement over Alloy 22 in the most aggressive conditions such as high temperature concentrated mineral acids.

In milder environments such as strong mineral acids at temperatures approximately below 80°C the corrosion rate of Alloy 22 may be undistinguishable from the newer generation Ni-Cr-Mo alloys

The corrosion behavior of Alloy 22 can be divided in an active corrosion mode and a passive corrosion mode.

In most industrial applications and for the proposed Yucca Mountain repository the corrosion behavior of Alloy 22 will be in the passive corrosion mode.

The passive corrosion rate of Alloy 22 decreases as the time increases due to the formation of an ever more impervious oxide film on the surface.

Even for laboratory time testing conditions, the corrosion rate of Alloy 22 in multi-ionic solutions that may simulate concentrated ground water is exceptionally low, in the order of only a few nanometers per year.

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